

FILE 'CAPLUS' ENTERED AT 17:22:33 ON 09 DEC 2001

L1 336 SEA MESOPOROUS MATERIAL
L2 49 SEA L1 AND GEL
L3 0 SEA L2 AND DOPANT
L4 1 SEA L2 AND DOPED
D L4 1
D L4 1 KWIC
L5 1 SEA L2 AND ALKOXIDES
D L5 1 KWIC
D L5 1 STD, KWIC, AB

FILE 'USPATFULL' ENTERED AT 17:26:45 ON 09 DEC 2001

L6 17 SEA MESO POROUS
L7 3 SEA L6 AND ALKOXIDE
D L7 1-3 STD, AB, KWIC
L8 12 SEA L6 AND GEL
D L8 1-12 STD, AB, KWIC .

L8 ANSWER 1 OF 12 USPATFULL
AN 2001:158540 USPATFULL
TI Process for nitration of aromatic compounds using a non-acid type
nitration process
IN Lee, Bon-Su, Inchon, Korea, Republic of
Chung, Kyoo-Hyun, Inchon, Korea, Republic of
Lee, Yoon-Sik, Seoul, Korea, Republic of
Kim, Young-Gyu, Seoul, Korea, Republic of
PA Inha University Foundation, Inchon, Korea, Republic of (non-U.S.
corporation)
PI US 6291726 B1 20010918
WO 9942433 19990826
AI US 2000-622285 20001018 (9)
WO 1998-KR285 19980918
20001018 PCT 371 date
20001018 PCT 102(e) date
PRAI KR 1998-5014 19980218
DT Utility
FS GRANTED
LN.CNT 440
INCL INCLM: 568/939.000
INCLS: 568/940.000; 568/932.000; 568/934.000
NCL NCLM: 568/939.000
NCLS: 568/932.000; 568/934.000; 568/940.000
IC [7]
ICM: C07C205-00
EXF 568/939; 568/940; 568/932; 568/934
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The nitration of aromatic compounds is achieved in high yield and
selectivity by using oxygen activated by an inorganic catalyst and
nitrogen dioxide. Since this process uses neither concentration nitric
nor sulfuric acids, the generation of spent waste acid does not occur.
Furthermore, the process does not encounter the problem of high costs
associated with the generation of ozone as in an alternative nitration
process. Since the solubility of oxygen in a reaction medium is
increased by using pressurized oxygen, nitrogen dioxide is activated by
a porous inorganic oxide and thus an aromatic compound(e.g., benzene) is
nitrated into a nitro compound (e.g., PhNO₂), the reaction rate is
significantly increased, and the recovery of reactants is easy due to
the insolubility of the catalyst.
SUMMANG. or more and a surface area of 100 m.sup.2 /g or more.
Catalysts fulfilling the two conditions include silica **gel**,
.gamma.-alumina, magnesium silicate, Zeolite, Kaoline, silicious earth.
For the Zeolite, MCM (**meso-porous** zeolite), sodium
mordenite, NaY and others are preferable, while Kieselguhr is not.
Moreover, Zeolite X or Y is better than. . .
SUMM . . . such a catalyst, ferric chloride is dissolved in a solvent, and
refluxed with an inorganic oxide catalyst such as silica **gel**.
The solution is then filtrated, washed and aged at a high temperature.
DETD After supplying 0.1 mol of benzene to an autoclave, also added to the
same were 2.0 g of silica **gel** (70-230 mesh, for column
chromatography) having a surface area of more than 100 m.sup.2 /g and a
micropore of 5. . .
DETD The same procedure as described in Example 1 was carried out, except
that silica **gel** (14-20 mesh, for desiccant) was used as a
catalyst. As a result, nitrobenzene was obtained at a yield of more. .
DETD 0.1 mol of benzene, 2 g of silica **gel** (14-20 mesh) and 0.15
mol of liquid nitrogen dioxide were poured into an autoclave. Next,
oxygen was injected at a. . .
DETD 0.1 mol of benzene, 2 g of silica **gel** (14-20 mesh) and 0.1 mol
of liquid nitrogen dioxide were poured into an autoclave. Next, oxygen

DET D was injected at a. . . 0.1 mol of toluene, 2 g of silica **gel** (14-20 mesh) and 0.2 mol of liquid nitrogen dioxide were poured into an autoclave. Next, oxygen was injected at a. . .

DET D 0.1 mol of chlorobenzene, 2 g of silica **gel** (14-20 mesh) and 0.2 mol of liquid nitrogen dioxide were poured into an autoclave. Next, oxygen was injected at a. . .

DET D 0.2 mol of toluene, 4 g of silica **gel** (14-20 mesh) and 0.9 mol of liquid nitrogen dioxide were poured into an autoclave. Next, oxygen was injected at a. . .

DET D 0.2 mol of benzene, 2 g of silica **gel** (14-20 mesh) and 0.9 mol of liquid nitrogen dioxide were poured into an autoclave. Next, oxygen was injected at a. . .

DET D . . . ferric chloride was dissolved in 100 ml of water and 25 ml of ethanol. To this, 12.5 g of silica **gel** (70-230 mesh, for column chromatography) was added and refluxed for 10 hours. After filtrating the solution, the catalyst was washed. . .

CLM What is claimed is:

3. The process according to claim 2, wherein said catalyst is selected from the group consisting of silica **gel**, .gamma.-alumina, magnesium silicate, Zeolite, Kaoline and silicious earth.

4. The process according to claim 3, wherein the Zeolite is **meso**-**porous** zeolite, sodium mordenite or NaY.

L8 ANSWER 2 OF 12 USPATFULL
AN 2001:67848 USPATFULL
TI Polyorganosiloxane catalyst
IN Okubo, Tsuneyuki, Osaka-fu, Japan
Matsu, Kazutoshi, Osaka-fu, Japan
Araki, Takao, Osaka-fu, Japan
Takai, Toshihiro, Hyogo-ken, Japan
PA Mitsui Chemicals, Inc., Japan (non-U.S. corporation)
PI US 6229037 B1 20010508
AI US 2000-543157 20000404 (9)
DT Utility
FS Granted
LN.CNT 746
INCL INCLM: 556/428.000
INCLS: 502/158.000; 502/168.000; 528/030.000; 568/727.000
NCL NCLM: 556/428.000
NCLS: 502/158.000; 502/168.000; 528/030.000; 568/727.000
IC [7]
ICM: C07F007-08
EXF 502/158; 502/168; 556/428; 528/30; 568/727
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The invention provides a polyorganosiloxane catalyst with both of a sulfonic acid group-containing hydrocarbon group and a mercapto group-containing hydrocarbon group, wherein the total amount of the sulfonic acid group-containing hydrocarbon group and the mercapto group-containing hydrocarbon group is within a range of 0.3 to 2.0 .mu.mol per unit surface area (1 m.sup.2) of the polyorganosiloxane catalyst. The catalyst of the invention is highly active for the reaction to produce bisphenol A from acetone and phenol, with the deterioration of the catalyst under extreme suppression.
SUMM These polyorganosiloxane catalysts are generally porous substances synthetically produced by so-called sol-**gel** process comprising hydrolysis of alkoxy silanes of different types and subsequent dehydration-condensation; and the porous substances comprise a macro-porous region of a pore size of 200 angstroms or more and a **meso**-**porous** region of a pore size within a range of 20 to 200 angstroms. The mean pore size in the **meso**-

porous region is generally 20 to 100 angstroms. So as to recover high catalytic activity, generally, the specific surface area is. . .

SUMM So-called sol-**gel** processes, including

SUMM For the sol-**gel** process of polyorganosiloxane, for example, a polyorganosiloxane with a high specific surface area of generally 500 to 600 m.² /g. . .

SUMM . . . substances in the reactor and the reaction conditions. The molded material satisfying the conditions includes for example active charcoal, silica **gel**, silica-alumina, alumina, molecular sieve, titania, silica-titania, zirconia, and zeolite. Among them, silica **gel** is particularly preferably used.

DETD . . . the phenylsulfonic acid group-containing ethoxysilane with impurities was used as a raw material for the sulfonic acid component for the sol-**gel** preparation of a polyorganosiloxane catalyst with sulfonic acid group-containing hydrocarbon group and mercapto group-containing hydrocarbon group.

DETD . . . added water of 15.1 g (838.89 mmol) over 30 minutes. To the resulting solution was charged a commercially available silica **gel** particle of 55 g (CARiACT Q-15 of particle size of 0.85 to 1.70 mm; manufactured by Fuji Silysia Chemicals, Co. . . stand for cooling, followed by distillation under reduced pressure with rotary evaporator, the resulting polyorganosiloxane was supported on the silica **gel** particle.

DETD . . . mixture of 5 ml of aqueous 28% ammonia and 35 ml of water was dropwise added to the resulting silica **gel**, followed by agitation at room temperature for 4 hours. For aging, the mixture was further agitated at 65.degree. C. for 3 days. The resulting mixture was again subjected to distillation under reduced pressure with a rotary evaporator. The silica **gel** particle was transferred into a 1000-ml beaker, followed by addition of 200 ml of 2N hydrochloric acid and agitation at room temperature for 30 minutes, so that the silica **gel** returned to the proton type. After separation by filtration, rinsing in 500 ml of ion exchange water was repeated twice, . . .

DETD . . . the resulting product was dried at 100.degree. C. under reduced pressure for 4 hours. By the aforementioned procedures, the silica **gel** particle supporting the polyorganosiloxane with the sulfonic acid group-containing hydrocarbon group and the mercapto group-containing hydrocarbon group was recovered at a yield of 84.6 g. The amount of the polyorganosiloxane supported on the silica **gel** particle was at 35% by weight in the supported catalyst 6. Additionally, the amount of the sulfonic acid group in. . .

DETD . . . catalyst 7 was recovered in the same manner as for the catalyst 6, except for no use of the silica **gel** particle as the carrier.

DETD . . . 8 was recovered in the same manner as for the catalyst 6, except that the particle size of the silica **gel** particle as the carrier was 75 to 500 .mu.m. The amount of the sulfonic acid group in the catalyst 8. . .

DETD Supported catalyst 9 was recovered in the same manner as for the catalyst 6, except that the silica **gel** particle as the carrier was replaced with silica-alumina. The amount of the sulfonic acid group in the catalyst 9 was. . .

DETD	1.14	0.38	0.21	1.14	0.86
solid acid(meq/g)					
Specific surface area(m. ² /g)	662	723	721	705	710
Average pore size of meso-porous	40	23	38	55	21
region (.ANG.)					
Pore volume of	0.66	0.39	0.70	1.00	0.39
					0.70

meso-porous

region (ml/g)

Active site 1.15 3.15 1.05 0.60 3.21 5.10

amount

(.mu.mol/m.sup.2)

TEOS; tetraethoxysilane

MMTMS; mercaptomethyltrimethoxysilane

MPTMS; mercaptopropyltrimethoxysilane

CLM What is claimed is:

6. The polyorganosiloxane-supported catalyst according to claim 5,

wherein the porous molded material is silica **gel** or
silica-alumina.

7. The polyorganosiloxane-supported catalyst according to claim 6,
wherein the porous molded material is silica **gel**.

L8 ANSWER 3 OF 12 USPATFULL

AN 2001:14414 USPATFULL

TI Supported catalyst useful for Friedel-Crafts reactions and process for
the preparation of aralkylated aromatic compounds using the catalyst

IN Choudhary, Vasant Ramchandra, Maharashtra, India

Jana, Suman Kumar, Maharashtra, India

Kiran, B-Phani, Maharashtra, India

PA Council of Scientific & Industrial Research, New Delhi, India (non-U.S.
corporation)

PI US 6180557 B1 20010130

AI US 1998-209834 19981211 (9)

PRAI IN 1998-238898 19980813

IN 1998-252698 19980826

DT Utility

FS Granted

LN.CNT 1382

INCL INCLM: 502/224.000

INCLS: 502/226.000; 502/227.000; 502/229.000; 502/231.000

NCL NCLM: 502/224.000

NCLS: 502/226.000; 502/227.000; 502/229.000; 502/231.000

IC [7]

ICM: B01J027-06

ICS: B01J027-125; B01J027-128; B01J027-135; B01J027-138

EXF 502/224; 502/226; 502/227; 502/229; 502/231

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A supported catalyst and a process for the preparation of the catalyst,
the catalyst containing mixed metal oxides or halides deposited on
porous catalyst carriers or supports, useful for heterogeneously or
solid catalyzed Friedel-Crafts reactions such as alkylation,
aralkylation, acylation, or aroylation of aromatic compounds in the
preparation of fine chemicals. The supported catalyst has high activity
for the Friedel-Crafts reactions when the aromatic ring activating
groups are present in the aromatic ring to be aralkylated, acylated, or
aroylated, and also when the ring activating group is absent or when
aromatic ring deactivating groups are present in the aromatic ring to
be aralkylated, acylated or aroylated, so that the reaction temperature is
low and/or the time for completing the reaction is short. The catalyst
can be separated and used repeatedly for catalytic reactions.

SUMM In the catalyst preparation process of this invention, the porous
catalyst carrier or support, S, is selected from micro- and/or
meso porous zeolites and zeolite-like materials,
synthetic and natural clays, silica **gel**, alumina and meso
and/or macroporous catalyst carriers containing SiO₂, Al₂O₃,
SiC, ZrO₂, HfO₂ or a mixture thereof, and more
preferably, S is selected from mesoporous zeolites such as high silica
MCM-41 and the like, silica **gel**, cation exchange clays such as

montmorillonite clay and the like, and chemically inert or sintered low surface area macroporous catalyst. . .

SUMM . . . wt %; and the preferred catalyst carrier or support, S, in the said catalyst may be selected from microporous silica **gel**, H-ZSM-5 or other pentasil (e.g. H-ZSM-9, H-ZSM-11, etc.) zeolite, mesoporous MCM-41 zeolite, montmorillonite clay, and meso- and/or macroporous catalyst. . .

DETD . . . 2.8 g gallium nitrate and 0.3 g indium nitrate, dissolved in 100 ml distilled water, on 50 g high silica **meso-porous** zeolite [prepared by the procedure described in the ref Choudhary et al., Proceeding of Indian Academy of Sciences, (Chemical Sciences). . .

DETD A supported catalyst: Ga_{sub.29.0} TiO_{sub.44.0} (10.8 wt %)/SiO_{sub.2} **gel** was prepared by impregnating a mixture of 14.0 g gallium nitrate and 0.5 g thallous nitrate, dissolved in 50 ml distilled water, on 50 g SiO_{sub.2} **gel** catalyst support (Fuji Davison, A-type, surface area 720 m².sup.2 g⁻¹) in powdered form, by incipient wetness technique, drying the impregnated mass. . .

CLM What is claimed is:

2. A supported solid catalyst as claimed in claim 1, wherein S is selected from micro- and/or **meso-porous** zeolites and zeolite-like materials, synthetic and natural clays, silica **gel**, alumina and mesoporous and macroporous catalyst carriers containing SiO_{sub.2}, Al_{sub.2}O_{sub.3}, SiC, ZrO_{sub.2}, HfO_{sub.2} or a mixture thereof.

7. A process as claimed in claim 6, wherein S is selected from micro- and/or **meso-porous** zeolites and zeolite-like materials, synthetic and natural clays, silica **gel**, alumina and **meso-porous** and macroporous catalyst carriers containing SiO_{sub.2}, Al_{sub.2}O_{sub.3}, SiC, ZrO_{sub.2}, HfO_{sub.2} or a mixture thereof.

L8 ANSWER 4 OF 12 USPATFULL
 AN 2000:131779 USPATFULL
 TI Catalyst composition free from noble metals
 IN Muller, Ulrich, Neustadt, Germany, Federal Republic of
 Schulz, Michael, Ludwigshafen, Germany, Federal Republic of
 Marosi, Laszlo, Ludwigshafen, Germany, Federal Republic of
 Harder, Wolfgang, Weinheim, Germany, Federal Republic of
 PA BASF Aktiengesellschaft, Ludwigshafen, Germany, Federal Republic of
 (non-U.S. corporation)
 PI US 6127307 20001003
 WO 9732866 19970912
 AI US 1998-142051 19980904 (9)
 WO 1997-EP1113 19970305
 19980904 PCT 371 date
 19980904 PCT 102(e) date
 PRAI DE 1996-19608493 19960305
 DT Utility
 FS Granted
 LN.CNT 719
 INCL INCLM: 502/162.000
 INCLS: 502/164.000; 502/167.000; 502/200.000; 502/208.000; 502/209.000;
 502/210.000; 502/211.000; 502/212.000; 502/213.000; 502/302.000;
 502/305.000; 502/325.000; 502/349.000; 502/353.000; 502/355.000
 NCL NCLM: 502/162.000
 NCLS: 502/164.000; 502/167.000; 502/200.000; 502/208.000; 502/209.000;
 502/210.000; 502/211.000; 502/212.000; 502/213.000; 502/302.000;
 502/305.000; 502/325.000; 502/349.000; 502/353.000; 502/355.000
 IC [7]
 ICM: B01J031-00

ICS: B01J027-24; B01J027-14; B01J027-192; B01J023-00
EXF 502/162; 502/164; 502/167; 502/208-213; 502/305-355; 502/200; 502/302
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB A noble metal-free catalyst composition is obtainable by

a) preparing an aqueous mixture comprising
i) a salt of at least one base metal selected from among the elements having atomic numbers 21-32, 39-42, 48-51, 57-75 and 81-83;
ii) phosphate ions; and
iii) at least one nitrogen source; and
b) evaporating the aqueous mixture obtained and drying the catalyst composition thus formed. The catalyst composition prepared can be used for producing hydrogen peroxide and for the epoxidation of olefins.
DETD The abovementioned, particularly preferred titanium silicates having an MFI pentasil structure are prepared by crystallizing a synthesis gel comprising water, a titanium source and silicon dioxide in an appropriate manner with addition of organic, nitrogen-containing compounds under hydrothermal. . .
CLM What is claimed is:
as claimed in claim 9 wherein the oxygen transferer is selected from among organometallic compounds, zeolites, zeolite analogs, aluminophosphates or **meso-porous** metal oxides which each comprise at least one metal selected from among Ti, V, Mo, W, Re and Ru.

L8 ANSWER 5 OF 12 USPATFULL
AN 2000:70416 USPATFULL
TI Process for the preparation of a micro-**meso porous** material with a high surface area and controlled distribution of the porosity
IN Perego, Carlo, Carnate, Italy
Carati, Angela, S. Giuliano Milanese, Italy
PA Eniricerche S.p.A., S. Donato Milanese, Italy (non-U.S. corporation)
PI US 6071485 20000606
AI US 1997-866057 19970530 (8)
PRAI IT 1996-MI1202 19960613
DT Utility
FS Granted
LN.CNT 367
INCL INCLM: 423/326.000
INCLS: 423/330.100; 423/707.000
NCL NCLM: 423/326.000
NCLS: 423/330.100; 423/707.000
IC [7]
ICM: C01B033-38
EXF 423/707; 423/326; 423/333; 423/330.1; 502/263; 502/240
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The present invention relates to a process for the preparation of a **micro-meso porous** material which comprises:

a) preparing a mixture starting from a tetraalkylorthosilicate, a C._{sub.3} -C._{sub.6} alkyl alcohol or dialcohol, a tetraalkylammonium hydroxide having the formula R._{sub.1} (R._{sub.2})._{sub.3} N-OH wherein R._{sub.1} is a C._{sub.3} -C._{sub.7} alkyl and R._{sub.2} is a C._{sub.1} or C._{sub.3} -C._{sub.7} alkyl, optionally in the presence of one or more metal compounds, in which the molar ratios are within the following ranges:

alcohol/SiO._{sub.2} < or equal to 20;

R._{sub.1} (R._{sub.2})._{sub.3} N--OH/SiO._{sub.2} =0.05-0.4;

H._{sub.2} O/SiO._{sub.2} =1-40;

metal oxides/SiO._{sub.2} =0-0.02;

b) subjecting this mixture to hydrolysis and subsequent gelation at a temperature of between 20.degree. C. and a temperature close to the boiling point of the alcohol or mixture of alcohols present;

c) subjecting the **gel** obtained to drying and calcination.

TI Process for the preparation of a **micro-meso porous** material with a high surface area and controlled distribution of the porosity

AB The present invention relates to a process for the preparation of a **micro-meso porous** material which comprises:

AB c) subjecting the **gel** obtained to drying and calcination.

SUMM The present invention relates to a process for the preparation of a **micro-meso porous** material having a high surface area and with a controlled distribution of the porosity.

SUMM c) subjecting the **gel** obtained to drying and calcination.

SUMM Patent application WO 91/11390 describes the possibility of obtaining a new group of **meso porous** alumino-silicates (called MCM-41) having an average pore diameter of between 20 .ANG. and 100 .ANG. and regularly organized in the. . .

SUMM . . . 20 .ANG. and mesopores, pores with a diameter of between 20 and 500 .ANG., these materials can be classified as **micro-meso porous**.

SUMM The solution obtained is then gelated by heating it to a temperature of between 50.degree. C. and 110.degree. C.; the **gel** obtained is then dried and calined to give the end-product.

SUMM Patent applications EP 95 200 093.3 and EP 96 104 680.2 also describe the possibility of preparing **micro-meso porous** metallo-silicates, called ERS-8, characterized by a narrow distribution of the pore dimensions with an average diameter of <40 .ANG., a. . .

SUMM . . . X-ray diffraction spectrum (XRD) from powders which has a widespread reflection at low angles, indicating a "short-range" ordering of the **micro-meso porous** structure.

SUMM This can be interpreted with the presence of a "short-range" ordering of the **micro-meso porous** structure, a structural correlation substantially limited only to the immediate neighbours.

SUMM The present invention therefore relates to a process for the preparation of a **micro-meso porous** material having a high surface area with a controlled distribution of the porosity, which basically comprises subjecting to hydrolysis, gelation. . .

SUMM c) subjecting the **gel** obtained to drying and calcination.

DETD After about 30 minutes of stirring, there is the formation of a transparent **gel** which is left to age for about 20 hours, is then dried in an oven at 120.degree. C. under vacuum. . .

DETD After about 30 minutes of stirring, there is the formation of a transparent **gel** which is left to age for about 20 hours, is then dried in an oven at 120.degree. C. under vacuum. . .

CLM What is claimed is:

1. A process for the preparation of a **micro-meso porous** material which comprises: a) preparing a mixture comprising a tetraalkyl-orthosilicate, at least one C._{sub.4} -C._{sub.6} alkyl alcohol, a dialcohol, or. . . C. and a temperature lower than the boiling point of the alcohol or mixture of alcohols present to obtain a **gel**; c) subjecting the **gel** obtained to drying and calcination; wherein said process is carried out in an open system.

4. A process for the preparation of a micro-**meso**
porous material which comprises: a) preparing a mixture
comprising: a tetraalkyl-orthosilicate, and at least one alcohol
selected from the group consisting. . . C. and a temperature lower
than the boiling point of the alcohol or mixture of alcohols present to
obtain a **gel**; c) subjecting the **gel** obtained to
drying and calcination; wherein said process is carried out in an open
system.

5. A process for the preparation of a micro-**meso**
porous material which comprises: a) preparing a mixture
comprising: a tetraalkyl-orthosilicate and at least one alcohol selected
from the group consisting. . . C. and a temperature lower than the
boiling point of the alcohol or mixture of alcohols present to obtain a
gel; c) subjecting the **gel** obtained to drying and
calcination; wherein said process is carried out in an open system.

6. A process for the preparation of a micro-**meso**
porous material which comprises: a) preparing a mixture
comprising: a tetraalkyl-orthosilicate and at least one alcohol selected
from the group consisting. . . this mixture to hydrolysis and
subsequent gelation at a temperature of between 25.degree. C. and
50.degree. C. to obtain a **gel**; c) subjecting the **gel**
obtained to drying and calcination; wherein said process is carried out
in an open system.

L8 ANSWER 6 OF 12 USPATFULL
AN 1999:159347 USPATFULL
TI Catalytic cracking catalyst and method for cracking a heavy oil
IN Itoh, Toshio, Sodegaura, Japan
PA Idemitsu Kosan Co., Ltd., Tokyo, Japan (non-U.S. corporation)
Petroleum Energy Center, Tokyo, Japan (non-U.S. corporation)
PI US 5997729 19991207
AI US 1998-8432 19980116 (9)
DT Utility
FS Granted
LN.CNT 720
INCL INCLM: 208/120.010
INCLS: 208/118.000; 208/119.000; 208/122.000; 502/063.000; 502/064.000;
502/068.000; 502/079.000
NCL NCLM: 208/120.010
NCLS: 208/118.000; 208/119.000; 208/122.000; 502/063.000; 502/064.000;
502/068.000; 502/079.000
IC [6]
ICM: C10G011-05
ICS: B01J029-06
EXF 502/63; 502/64; 502/68; 502/79; 208/120.01; 208/118; 208/119; 208/122
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB A catalytic cracking catalyst, which comprises (1) a zeolite, (2) a
silica.cndot.alumina or an alumina and (3) a kaolin, has a pore diameter
distribution having a peak at a pore diameter of 450 to 3,000 .ANG., and
has 40 to 75% of a pore volume of pores of not less than 200 .ANG. and
less than 2,000 .ANG. in pore diameter and 5 to 45% of a pore volume of
pores of not less than 2,000 .ANG. and less than 18,000 .ANG. in pore
diameter, both based on a pore volume of pores of 40 to 18,000 .ANG. in
pore diameter.
SUMM . . . literature refers to the sizes of pores of catalytic cracking
catalysts. Japanese Patent Application Unexamined Publication No.
6-25675 (1994) discloses **meso-porous** cracking
catalysts, which, however, have a pore distribution with a peak around
150 to 350 .ANG., and cannot crack tower. . .
SUMM . . . can be produced, for example, by a method which comprises

spray-drying a slurry containing (a) a zeolite, (b) a silica.cndot.alumina **gel** or an alumina **gel** and (c) a kaolin, or spray-drying the slurry and then calcining, wherein the (b) silica.cndot.alumina **gel** or the (b) alumina **gel** is used in such an amount that when the catalytic cracking catalyst is calcined to give a calcined product, the. . . product contains 2 to 50 wt % of a silica.cndot.alumina or an alumina which is derived from the (b) silica.cndot.alumina **gel** or the (b) alumina **gel**, respectively.

SUMM wherein the (b) silica.cndot.alumina **gel** or the (b) alumina **gel** is used in such an amount that when the catalytic cracking catalyst is calcined to give a calcined product, the. . . product contains 2 to 50 wt % of a silica.cndot.alumina or an alumina which is derived from the (b) silica.cndot.alumina **gel** or the (b) alumina **gel**, respectively.

SUMM . . . method which comprises spray-drying a slurry containing (a) a zeolite, (b') a silica.cndot.alumina which is obtained by calcining a silica.cndot.alumina **gel**, or an alumina which is obtained by calcining an alumina **gel**, and (c) a kaolin, or spray-drying the slurry and then calcining, wherein the silica.cndot.alumina **gel** or the alumina **gel**

SUMM wherein the silica.cndot.alumina **gel** or the alumina **gel** is used in such an amount that when the catalytic cracking catalyst is calcined to give a calcined product, the. . . calcined product contains 2 to 50 wt % of a silica.cndot.alumina or an alumina which is derived from the silica.cndot.alumina **gel** or the alumina **gel**, respectively.

SUMM Herein, the silica.cndot.alumina or the alumina derived from the silica.cndot.alumina **gel** or the alumina **gel**, respectively, means only the silica.cndot.alumina derived from the starting silica.cndot.alumina **gel** or the alumina derived from the starting alumina **gel**, and does not include other silica.cndot.alumina or other alumina derived from other components, such as zeolites.

SUMM Raw materials of the silica.cndot.alumina **gel** or the alumina **gel**, which is used in the methods of the present invention and is to be a matrix of the catalytic cracking. . . colloidal silica, and an alumina source, such as aluminum sulfate or sodium aluminate. For example, the production of the silica.cndot.alumina **gel** or the alumina **gel** from these raw materials may be performed as follows.

SUMM The silica.cndot.alumina **gel** or the alumina **gel** has a silica/alumina ratio of 0/100 to 80/20 (wt/wt), preferably 0/100 to 70/30 (wt/wt), more preferably 10/90 to 70/30 (wt/wt). . . .

SUMM The following is an example of the preparation of a silica.cndot.alumina **gel**. JIS No. 3 water glass (content of silica as SiO₂ : 28 wt %) is diluted with ion-exchanged water to. . . .

SUMM . . . the resulting filtered cake is washed with ion-exchanged water. For the production of catalytic cracking catalysts, the filtered cake (silica.cndot.alumina **gel** or alumina **gel**) containing water may be used as it is, or after drying at 60 to 200.degree. C. for 1 to 12. . . .

SUMM When calcined under the above conditions, the silica.cndot.alumina **gel** or the alumina **gel** to be used in the method of the present invention should have a pore diameter distribution having a peak at. . . .

SUMM According to the methods of the present invention, a zeolite, the silica.cndot.alumina **gel** or alumina **gel** obtained as above and a kaolin, or a zeolite, a silica.cndot.alumina or alumina, which is obtained by calcining the silica.cndot.alumina **gel** or alumina **gel** obtained as above, and a kaolin, are mixed with ion-exchanged water, to form a slurry with a solid concentration of. . . .

SUMM . . . to 50 wt %, preferably 5 to 30 wt %, of a silica.cndot.alumina or an alumina derived from the silica.cndot.alumina **gel** or the alumina **gel**, preferably 10 to 50 wt %, more preferably 20 to 40 wt %, of a kaolin, 0 to 20 wt. . . .

DETD (1) Preparation of a silica.cndot.alumina **Gel**

(silica/alumina=50 wt/50 wt, peak of pore diameter distribution: 300 .ANG.)

DETD . . . aqueous aluminum sulfate solution were added alternately thereto each by 100 ml portions for three times, to prepare a silica.cndot.alumina **gel**. When the aqueous water glass solution was added, the mixture was adjusted to pH 10, and when the aqueous aluminum. . .

DETD After the resulting mixture was adjusted to pH 8, the reaction product was filtered off to obtain a crude silica.cndot.alumina **gel** as a filtered cake. The filtered cake was dispersed in 1 liter of 1 wt % aqueous ammonium nitrate solution. . . was repeated four times, the filtered cake was washed with ion-exchanged water and filtered off, to obtain a washed silica.cndot.alumina **gel**. When the washed silica.cndot.alumina **gel** was dried at 80.degree. C. for 6 hours and then calcined at 800.degree. C. for 3 hours, the calcined product. . .

DETD 10 wt % (based on the weight after calcination; the same shall apply hereinafter) of the silica.cndot.alumina **gel** obtained above, 30 wt % of USY zeolite (Trade name: HSZ-330HUA, produced by Toso Co., Ltd.), 40 wt % of. . . Co., Ltd.) and 10 wt % of silica sol were added to ion-exchanged water, to give a slurry. The silica.cndot.alumina **gel**, the zeolite and the kaolin had been previously ground to an average particle diameter of 0.9 .mu.m.

DETD . . . of Example 1 was repeated with the exceptions that in step (2) (Preparation of a Catalytic Cracking Catalyst), the silica.cndot.alumina **gel** prepared in step (1) of Example 1 was dried at 80.degree. C. for 6 hours and then calcined at 800.degree.. . .

DETD . . . aqueous aluminum sulfate solution in sequence, and the mixture was then adjusted to pH 8, to give a crude silica.cndot.alumina **gel**. Thereafter, the same procedure as in step (1) of Example 1 was repeated. When the washed silica.cndot.alumina **gel** prepared in this example was calcined, the calcined product had a pore diameter distribution having a peak at 100 .ANG..

DETD Thereafter, steps (2) and (3) of Example 1 were repeated with the exception that the silica.cndot.alumina **gel** obtained as above was used in place of the silica.cndot.alumina **gel** prepared in Example 1.

DETD . . . repeated with the exception that in step (2) (Preparation of a Catalytic Cracking Catalyst), 5 wt % of the silica.cndot.alumina **gel** prepared in Example 1, 30 wt % of USY zeolite, 40 wt % of kaolin, 12.5 wt % of silica. . .

DETD The procedure of Example 1 was repeated with the exception that the silica.cndot.alumina **gel** that was produced in Example 3 and had a pore diameter peak at 100 .ANG. was used in place of the silica.cndot.alumina **gel** produced in Example 1, and the USY zeolite and the kaolin had been ground to an average particle diameter of. . .

DETD . . . solution and the aqueous aluminum sulfate solution alternately each by 50 ml portions for six times, to prepare an alumina **gel**. When the aqueous sodium aluminate solution was added, the mixture was adjusted to pH 10 or above, and when the. . .

DETD The obtained alumina **gel** was treated in the same manner as in Example 1, to obtain an alumina **gel** with a decreased sodium content. When the alumina **gel** was dried at 80.degree. C. for 3 hours and then calcined at 800.degree. C. for 3 hours, the calcined product. . .

DETD The alumina **gel** was dried at 80.degree. C. for 3 hours and then calcined at 500.degree. C. for 3 hours to obtain alumina. . .

DETD . . . and the aqueous aluminum sulfate solution alternately each by 50 ml portions for 8 times, to give a crude silica.cndot.alumina **gel**.

DETD . . . wt % aqueous sodium hydroxide solution. Then the same procedure as in Example 1 was repeated. When the obtained silica.cndot.alumina

DET D . . . **gel** was calcined, the calcined product had a pore diameter distribution having a peak at 600 .ANG..

DET D . . . cracking catalyst was produced in the same manner as step (2) of Example 1, with the exception that the silica.cndot.alumina **gel** obtained as above, zeolite and kaolin which were ground to an average particle diameter of 2.2 .mu.m were used.

DET D . . . cracking catalyst was produced in the same manner as step (2) of Example 1 with the exception that the silica.cndot.alumina **gel** used in Example 1 was not used, but 30 wt % of the USY zeolite, 40 wt % of the. . .

DET D . . . in the same manner as step (2) of Example 1 with the exception that 40 wt % of the silica.cndot.alumina **gel** produced in Example 1, 30 wt % of the USY zeolite, 20 wt % of the kaolin, 5 wt %. . .

DET D . . . sulfate solution, which were prepared in Example 1, were added thereto simultaneously with continuous stirring, to prepare a crude silica.cndot.alumina **gel**. The crude silica.cndot.alumina **gel** was washed in the same manner as in Example 1. When the washed silica.cndot.alumina **gel** was dried at 80.degree. C. for 3 hours and then calcined at 800.degree. C. for 3 hours, the calcined product. . .

DET D . . . cracking catalyst was produced in the same manner as step (2) of Example 1 with the exception that the silica.cndot.alumina **gel** prepared above was used in place of the silica.cndot.alumina **gel** prepared in Example 1.

DET D . . . in Example 1 were poured thereto alternately each by 50 ml portions for 8 times, to prepare a crude silica.cndot.alumina **gel**. Then the same procedure as in Example 1 was repeated. When the crude silica.cndot.alumina **gel** was washed, dried and calcined in the same manner as in Example 1, the calcined product had a pore diameter. . .

DET D . . . catalyst was produced in the same manner as step (2) of Example 1 with the exceptions that the washed silica.cndot.alumina **gel** obtained above was used in place of the silica.cndot.alumina **gel** prepared in Example 1, and that the USY zeolite and the kaolin, which were the same as those used in. . .

DET D . . . and the aqueous aluminum sulfate solution alternately each by 50 ml portions for 8 times, to prepare a crude silica.cndot.alumina **gel**. When the aqueous water glass solution was added, the mixture was adjusted to pH 10 or above, and when the. . . 5 wt % aqueous ammonia. Then the same procedure as in Example 1 was repeated to prepare a washed silica.cndot.alumina **gel**. When the washed silica.cndot.alumina **gel** was dried and calcined in the same manner as in Example 1, the calcined product had a pore diameter distribution. . . carried out in the same manner as steps (2) and (3) of Example 1 with the exception that the silica.cndot.alumina **gel** prepared as above was used in place of the silica.cndot.alumina **gel** prepared in Example 1.

DET D . . . and the aqueous aluminum sulfate solution alternately each by 50 ml portions for 10 times, to prepare a crude silica.cndot.alumina **gel**.

DET D . . . 5 wt % aqueous ammonia. Then the same procedure as in Example 1 was repeated to prepare a washed silica.cndot.alumina **gel**. When the washed silica.cndot.alumina **gel** was dried and calcined in the same manner as in Example 1, the calcined product had a pore diameter distribution. . .

DET D . . . cracking catalyst was produced in the same manner as step (2) of Example 1 with the exceptions that the silica.cndot.alumina **gel** prepared as above was used in place of the silica.cndot.alumina **gel** prepared in Example 1, and that the zeolite and the kaolin were ground to an average particle diameter of 2.9. . .

DET D . . . diameter where the

300 300 100 300 100 500 100 600
pore diameter distribution of
a calcined product of a
silica .multidot. alumina **gel** or a
silica .multidot. alumina has a peak
(.ANG.)
(.DELTA.V/.DELTA.logD)/350 .ANG.
0.00909
0.004
0.009
0.004
0.005
0.010
0.007

(cc/g .multidot. .mu.m.ANG.)

Content of silica .multidot.. 5 6
DETD

Pore diameter where the
-- 300 30 500 600 700
pore diameter distribution of
a calcined product of a
silica .multidot. alumina **gel** or a
silica .multidot. alumina has a peak
(.ANG.)
(.DELTA.V/.DELTA.logD)/350 .ANG.
--
0.009
0.002
0.015
0.002
0.005

(cc/g .multidot. .mu.m.ANG.)

Content of silica .multidot. alumina
. . . .

CLM What is claimed is:

. . . of producing the catalytic cracking catalyst of claim 1, comprising
spray-drying a slurry containing (a) a zeolite, (b) a silica-alumina
gel or an alumina **gel** and (c) a kaolin, and then
calcining the spray-dried slurry, wherein the silica-alumina **gel**
or the alumina **gel** has a silica/alumina weight ratio of 0/100
to 80/20, and when calcined by itself, gives a calcined product which
has. . . of the peak divided by 350 .ANG. has a value of 0.0030 to
0.0130 cc/g.multidot..mu.m .ANG., and wherein the silica-alumina
gel or the alumina **gel** is used in such an amount that
when the catalytic cracking catalyst is calcined to give a calcined
product, the calcined product contains 2 to 50 wt % silica-alumina or
alumina which is derived from the silica-alumina **gel** or the
alumina **gel**, respectively.

4. The method of claim 3, wherein the (a) zeolite, the silica-alumina
gel or the alumina **gel** and the (c) kaolin have been
ground to 0.2 to 5 .mu.m in diameter.

. . . claim 1, comprising spray-drying a slurry containing (a) a zeolite,
(b) a silica-alumina which is obtained by calcining a silica-alumina
gel, or an alumina which is obtained by calcining an alumina
gel, and (c) a kaolin, and then calcining the spray-dried
slurry, wherein the silica-alumina **gel** or the alumina
gel has a silica/alumina weight ratio of 0/100 to 80/20, and,
when calcined by itself, gives a calcined product which has. . . of
the peak divided by 350 .ANG. has a value of 0.0030 to 0.0130
cc/g.multidot..mu.m .ANG., and wherein the silica-alumina **gel**

or the alumina **gel** is used in such an amount that when the catalytic cracking catalyst is calcined to give a calcined product, the calcined product contains 2 to 50 wt % silica-alumina or alumina which is derived from the silica-alumina **gel** or the alumina **gel**, respectively.

13. The method of claim 12, wherein the (a) zeolite, the (b) silica-alumina **gel** or alumina **gel** and the (c) kaolin have been ground to 0.2 to 5 .mu.m in diameter.

L8 ANSWER 7 OF 12 USPATFULL
AN 1999:117149 USPATFULL
TI Transparent bulk silica porous material with uniform pore size and distribution
IN Sugimoto, Noriaki, Nagoya, Japan
Inagaki, Shinji, Nagoya, Japan
Fukushima, Yoshiaki, Aichi-ken, Japan
Hioki, Tatsumi, Nagoya, Japan
Ogawa, Makoto, Mitaka, Japan
PA Kabushiki Kaisha Toyota Chuo Kenkyusho, Aichi-ken, Japan (non-U.S. corporation)
PI US 5958577 19990928
AI US 1998-3461 19980106 (9)
PRAI JP 1997-14634 19970110
DT Utility
FS Granted
LN.CNT 507
INCL INCLM: 428/333.000
INCLS: 428/312.600; 428/316.600; 428/446.000; 423/335.000; 423/338.000;
423/339.000
NCL NCLM: 428/333.000
NCLS: 423/335.000; 423/338.000; 423/339.000; 428/312.600; 428/316.600;
428/446.000
IC [6]
ICM: B32B003-12
EXF 423/335; 423/338; 423/339; 428/312.6; 428/316.6; 428/333; 428/446
AB The method for producing a bulk silica porous material is presented. The obtained material has a large crystal size serving to reduce light scattering, and uniform and adjustable pore size. This can be utilized for optical and electronic functional materials. The method comprises the steps of:
forming a silica/surfactant composite by mixing starting materials containing an alkoxy silane, water and a surfactant and allowing the starting materials to react;
maturing said silica/surfactant composite by allowing the same to stand in a closed container to effect the development of a silica network structure and the formation of porous structure in said composite;
drying said matured silica/surfactant composite for removing the solvent and for condensation of said matured silica/surfactant composite; and,
sintering said condensed silica/surfactant composite to remove the surfactant to obtain a silica porous material.
SUMM . . . 1 to 10 nm and the pore size distributing in an extremely narrow range is referred especially to as a **meso porous** material.
SUMM Such a **meso porous** material is used as a material for separation and adsorption of gases and solutions because of its substantially uniform and. . .
SUMM The **meso porous** materials described above are

produced, for example, by the ion exchange between Kanemite which is one of lamellar silicates and a surfactant (Japanese Laid-Open Patent Publication 4-238810) or by the hydrothermal synthesis from silica **gel** and a surfactant in an air-tight pressure vessel.

SUMM However, only a **meso porous** material having a small crystal size can be obtained by a conventional method.

SUMM Accordingly, when such a **meso porous** material is used as an optical functional material, undesirable light scattering leads to only a limited range of application.

SUMM When such a **meso porous** material is used as an electronic functional material, poor processability of the **meso porous** material leads to the requirement of a special method for obtaining an electrode contact, resulting in inconvenience for handling.

SUMM Thus, a **meso porous** material obtained by a conventional method is difficult to be utilized as an optical or electronic functional material.

SUMM . . . or atomic cluster as large as 1 mm or larger, which can not be achieved by a conventional zeolite or **meso porous** material, is obtained.

SUMM . . . condition (i.e., in the presence of water and heat) into an organic micelle, which is subsequently crystallized to form a **meso porous** material. As a result, the organic micelle described above is in the form of a hexagonal crystal lattice, whereby representing. . .

SUMM On the other hand, in a conventional **meso porous** material, the organic micelles are aligned in the structure in a hexagonal system. However, since the silica component becomes larger. . .

DETD . . . water added as described above is less than 0.5 moles, the hydrolysis of the alkoxy silane is not sufficient to effect **gel** formation, and a bulk silica porous material may not be obtained. When the amount of the water exceeds 10 moles, . . .

DETD . . . size distribution curve mentioned above is obtained by plotting the value (dV/dD) obtained by differentiating the pore volume of a **meso porous** material by the pore size (D) versus the pore size (D) (FIG. 2).

L8 ANSWER 8 OF 12 USPATFULL
AN 1998:115907 USPATFULL
TI Process for the selective oxidation of hydrocarbons and their derivatives
IN Alive, Keshavaraja, National Chemical Laboratory, Pune-411008, Maharashtra, India
Venkataraman, Ramaswamy Arumugamangalam, National Chemical Laboratory, Pune-411008, Maharashtra, India
Paul, Ratnasamy, National Chemical Laboratory, Pune-411008, Maharashtra, India
PI US 5811599 19980922
AI US 510291& 19950802 (8)
PRAI IN 1722/Del/94 19941230
DT Utility
FS Granted
LN.CNT 493
INCL INCLM: 568/771.000
INCLS: 568/719.000; 568/741.000; 568/780.000; 568/803.000; 568/836.000;
549/518.000
NCL NCLM: 568/771.000
NCLS: 549/518.000; 568/719.000; 568/741.000; 568/780.000; 568/803.000;
568/836.000
IC [6]
ICM: C07C037-00
EXF 568/719; 568/741; 568/780; 568/771; 568/803; 568/836; 549/518
CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A process for the oxidation of hydrocarbons and their derivatives having the general formula RX, wherein R is from n-alkyl, iso-alkyl, benzyl, cyclohexyl, mono, di or tricyclic aryl, or alkenic groups and X is selected from H, OH or Cl to compounds having formula R.sup.1 XY wherein R.sup.1 =(R--H), X has the meaning defined as above and Y=OH; which comprises of reacting the appropriate hydrocarbon or it's derivative of the formula RX where R and X have the meaning given above, with a solution of aqueous hydrogen peroxide at a temperature in the range of 10.degree.-100.degree. C. in the presence of an amorphous titanium-silicate catalyst having molar chemical composition in terms of the anhydrous oxides of TiO₂ : (5-400) SiO₂, having an average micropore radius between 10 and 40 .ANG., an absorption band around 220 nm in the ultraviolet region, a band around 960 cm⁻¹ in the infrared region, interatomic vectors around 1.6-1.7, 2.7-2.8, 4.1-4.2 and 5.0-5.2 .ANG. in the radial electron density distribution and absence of any line in the x-ray diffraction pattern; and isolating the resultant products of the oxidation reaction by conventional methods.

DETD . . . Water was then added in a controlled manner to the above mixture in such a way that a clear transparent **gel** was obtained. The material was aged at 40.degree. C. for 24 hours after which the H₂O and alcohol were removed by vacuum treatment. The resultant semi-dry **gel** was dried in an oven at 110.degree. C. for 24 hours and further calcined at 400.degree. C. for 24 hours. . . .

CLM What is claimed is:

. . . 960 cm⁻¹ in the infrared region of the absorption spectrum, (c) having an average micropore radius below 10 .ANG. and **meso-porous** radius below 40 .ANG., (d) presence of a sharp absorption band around 200 nanometers in the ultraviolet region of the. . . .

L8 ANSWER 9 OF 12 USPATFULL
AN 1998:98585 USPATFULL
TI Micro-**meso porous** amorphous titanium silicates and a process for preparing the same
IN Alive, Keshavaraja, National Chemical Laboratory, Pune-411008, Maharashtra, India
Vedanayaki, Ramaswamy, National Chemical Laboratory, Pune-411008, Maharashtra, India
Venkataraman, Ramaswamy Arumugamangalam, National Chemical Laboratory, Pune-411008, Maharashtra, India
Paul, Ratnasamy, National Chemical Laboratory, Pune-411008, Maharashtra, India
PI US 5795555 19980818
AI US 1995-510574 19950802 (8)
PRAI IN 1994-150894 19941124
DT Utility
FS Granted
LN.CNT 462
INCL INCLM: 423/326.000
INCLS: 423/598.000
NCL NCLM: 423/326.000
NCLS: 423/598.000
IC [6]
ICM: C01B033-20
EXF 423/326; 423/598
CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A novel micro-**meso porous** amorphous titanium silicates having a molar composition in terms of the anhydrous oxides of TiO₂ : (5-400) SiO₂, the said titanium silicates being further characterised by (a) the absence of any line corresponding to individual oxides of titanium and silicon in its x-ray diffraction pattern, (b) the presence of interatomic vectors around 1.6-1.7, 2.7-2.8, 4.1-4.2 and 5.0-5.2 .ANG. in the radial electron density distribution, (c) an absorption band around 220 nm in the ultraviolet region corresponding to

tetrahedral Si--O--Ti linkage, (d) an absorption band around 960 cm.sup.-1 in infrared region corresponding to tetrahedral Si--O--Ti linkage and (e) a bimodal pore size distribution with the first peak width maximum at a value less than 10 .ANG. and the second peak width maximum between 10 and 100 .ANG., respectively; and a process of preparing said micro-**meso porous** amorphous titanium silicates without any nitrogenated organic base and at a neutral pH of around 7.

TI Micro-**meso porous** amorphous titanium silicates and a process for preparing the same

AB A novel micro-**meso porous** amorphous titanium silicates having a molar composition in terms of the anhydrous oxides of TiO₂ : (5-400) SiO₂, the said titanium. . . 10 .ANG. and the second peak width maximum between 10 and 100 .ANG., respectively; and a process of preparing said micro-**meso porous** amorphous titanium silicates without any nitrogenated organic base and at a neutral pH of around 7.

SUMM This invention relates to a process for the preparation of micro-**meso porous** amorphous titanium silicates. More particularly, it relates to novel micro-**meso porous** titanium silicates possessing catalytic properties and a method for their preparation.

SUMM The present invention relates to a novel family of stable synthetic micro-**meso porous** amorphous titanosilicates, identified as MMATS, and possessing specific characteristic which distinguish them from other titanosilicates of prior art.

SUMM . . . invention also relates to a method for preparing the synthetic titanium silicates with six distinctive features described hereinabove i.e. a micro-**meso porous** amorphous titanium silicates having a molar composition in terms of the anhydrous oxides of TiO₂ : (5-400) SiO₂ and also comprising. . .

DRWD FIG. 1 is a graphical representation of the radial electron density distribution for a crystalline titano-silicate, TS-1 and for a micro-**meso porous** amorphous titanium silicate, MMATS-1.

DRWD . . . a graphical comparison of the pore size distribution of a crystalline titano-silicate, TS-1 with the pore size distribution of a micro-**meso porous** amorphous titanium silicate, MMATS-1.

DETD Accordingly, in the present invention there is provided a process for the preparation of amorphous micro-**meso porous** titanosilicates which comprises:

DETD . . . removing water from sol D by allowing it to age at a temperature below 90.degree. C. to obtain the solid **gel** E and drying this **gel** at a temperature ranging from 90.degree. to 120.degree. C. and calcining at a temperature above 300.degree. C. to form micro-**meso porous** amorphous titano silicate.

DETD . . . of silicon, the ethoxides, propoxides, butoxides or any of their combinations may be used. After removal of water from the **gel**, the material may be dried at a temperature above 100.degree. C. and further calcined at a temperature between 300.degree. and. . .

DETD An amorphous micro-**meso porous** material containing titanium and silicon as oxides was synthesised from the respective alkoxides using an alcohol and water. 7.5 gm. . . water was then added, while stirring continued. The mixture was kept at 50.degree. C. for 36 hrs. to form a **gel** which was then dried at 110.degree. C. for 24 hrs. This was further calcined at 450.degree. C. for 12 hrs..

DETD . . . water was then added while the temperature was raised to 65.degree. C. and kept for 36 hrs. to form a **gel** which was then dried at 110.degree. C. for 24 hrs. The sample was finally calcined at 500.degree. C. for 12. . .

DETD . . . water was then added while the temperature was raised to

65.degree. C. and kept for 36 hrs to form a **gel** which was then dried at 110.degree. C. for 24 hrs. The sample was finally calcined at 500.degree. C. for 12. . .

CLM What is claimed is:

1. A process for the preparation of **micro-meso porous** amorphous titanium silicates having a molar composition in terms of the anhydrous oxides of TiO₂ : (5-400) SiO₂, the said titanium. . . water from the sol D by allowing it to age at a temperature below 90.degree. C. to obtain a solid **gel** E and drying this **gel** at a temperature ranging from 90.degree. to 120.degree. C. and calcining the dried **gel** at a temperature above 300.degree. C. to form the **micro-meso porous** amorphous titanium silicate.

L8 ANSWER 10 OF 12 USPATFULL

AN 1998:70988 USPATFULL

TI Adsorption heat pump

IN Inagaki, Shinji, Nagoya, Japan

Yamada, Yuri, Nagoya, Japan

Fukushima, Yoshiaki, Aichi-ken, Japan

Hasatani, Masanobu, Nagoya, Japan

Watanabe, Fujio, Owariasahi, Japan

PA Kabushiki Kaisha Toyota Chuo Kenkyuso, Aichi-ken, Japan (non-U.S. corporation)

PI US 5768910 19980623

AI US 1996-738113 19961025 (8)

PRAI JP 1995-279318 19951026

JP 1995-321783 19951211

DT Utility

FS Granted

LN.CNT 1094

INCL INCLM: 062/480.000
INCLS: 502/407.000; 502/527.000

NCL NCLM: 062/480.000
NCLS: 502/407.000

IC [6]

ICM: F25B017-08

ICS: B01J020-10

EXF 062/476; 062/480; 062/101; 502/407; 502/527

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB An adsorption heat pump includes a working fluid, an adsorption-desorption unit, and an evaporation-condensation unit connected with the adsorption-desorption unit. The adsorption-desorption unit adsorbs and desorbs vapor resulting from the working fluid, and includes an adsorbent being a porous substance. The porous substance has pores, and exhibits a pore diameter distribution curve having a maximum peak falling in a pore diameter range of from 1 to 10 nm. The pores in the diameter range of .+- .40% of pore diameter at the maximum peak have pore volume not less than 60% of a whole volume of the porous substance. The evaporation-condensation unit evaporates and condenses the working fluid. The adsorption heat pump can be operated by a low-temperature heat source, and can exhibit a large pumping temperature difference regardless of its small size.

SUMM Silica **gel**, activated alumina, zeolite, and activated carbon have been examined for the application to adsorbents for adsorption heat pumps. These adsorbents. . . be enlarged. Activated alumina also suffers from problems similar to those of zeolite. On the other hand, in a silica **gel**-water system, or an activated carbon-water system, adsorbents can be recovered by a relatively low-temperature heat source of 100.degree. C. or. . .

DRWD FIG. 7 illustrates the X-ray diffraction patterns which were exhibited by **meso-porous** substances prepared in the First

Preferred Embodiment of the present invention;
DRWD FIG. 8 is a picture on the pore-structure of one of the **meso-porous** substances prepared in the First Preferred Embodiment of the present invention, the picture which was taken by a transmission electron microscope to show a pore diameter distribution of the **meso-porous** substance;

DRWD FIG. 9 illustrates the nitrogen adsorption isotherms which were exhibited by the **meso-porous** substances prepared in the First Preferred Embodiment of the present invention;

DRWD FIG. 10 is a graph for illustrating the pore diameter distribution curves which were exhibited by the **meso-porous** substances prepared in the First Preferred Embodiment of the present invention, the graph which is determined based on FIG. 9;

DRWD FIG. 11 illustrates the nitrogen adsorption isotherms which were exhibited by comparative examples (e.g., activated carbon, silica **gel**, and zeolite);

DRWD FIG. 13 is a graph for illustrating the pore diameter distribution curves which were exhibited by **meso-porous** substances prepared in the Second Preferred Embodiment of the present invention, the **meso-porous** substances which were densely packed;

DRWD FIGS. 14A to D are graphs for illustrating the water-vapor adsorption of a "FSM/10" **meso-porous** substance in the Second Preferred Embodiment of the present invention, subjected to different preliminary treatments, wherein;

DRWD FIG. 14A illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the "FSM/10" **meso-porous** substance subjected to the first preliminary treatment at 25.degree. C. for 3 hours;

DRWD FIG. 14B illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the "FSM/10" **meso-porous** substance subjected to the second preliminary treatment at 25.degree. C. for 3 hours;

DRWD FIG. 14C illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the "FSM/10" **meso-porous** substance subjected to the third preliminary treatment at 25.degree. C. for 3 hours; and

DRWD FIG. 14D illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the "FSM/10" **meso-porous** substance subjected to the fourth preliminary treatment at 70.degree. C. for 3 hours;

DRWD FIGS. 15A and B are graphs for illustrating the water-vapor adsorption isotherms of a "FSM/16" **meso-porous** substance in the Second Preferred Embodiment of the present invention, subjected to different preliminary treatments, wherein;

DRWD FIG. 15A illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the "FSM/16" **meso-porous** substance subjected to the first preliminary treatment at 300.degree. C. for 3 hours; and

DRWD FIG. 15B illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the "FSM/16" **meso-porous** substance subjected to the second preliminary treatment at 25.degree. C. for 3 hours; and

DRWD FIGS. 16A to C are graphs for illustrating the water-vapor adsorption isotherms of a type "A" silica **gel**, subjected to different preliminary treatments, wherein;

DRWD FIG. 16A illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the type "A" silica **gel** subjected to the first preliminary treatment at 80.degree. C. for 3 hours;

DRWD FIG. 16B illustrates a water-vapor adsorption isotherm at an adsorption temperature 25.degree. C. of the type "A" silica **gel** subjected to the second preliminary treatment at 25.degree. C. for 4 hours; and

DRWD FIG. 16C illustrates a water-vapor adsorption isotherm at an adsorption

temperature 20.degree. C. of the type "A" silica **gel** subjected to the third preliminary treatment at 25.degree. C. for 4 hours. . . . pump can utilize a porous substance as the adsorbent. As for the porous substance, it is possible to utilize a **meso-porous** substance. The **meso-porous** substance can be synthesized by reacting a layer silicate with a surfactant (See T. Yanagisawa et al., Bull. Chem. Soc. Japan., 63, pp. 988-992 (1990) incorporated herein by reference.). The **meso-porous** substance has a structure which comprises silicate sheets, and cylindrical pores. The silicate sheets are bent periodically (e.g., at intervals. . . and are arranged periodically in the clearances between the silicate sheets. According to an X-ray diffraction pattern exhibited by the **meso-porous** substance, at least one peak including a maximum intensity peak can be observed at positions which correspond to the d-spacing. . . .

DETD In addition to the aforementioned porous substance, there is provided a **meso-porous** molecular sieve (MCM-41) which is produced by using the micelle texture of a surfactant as a template (See Kresge et. . . honeycomb-shaped cross-section as well. However, in the cellular walls, the MCM-41 has a different structure from that of the aforementioned **meso-porous** substance. According to an X-ray diffraction pattern exhibited by the MCM-41, at least one peak can be observed at positions. . . .

DETD On the other hand, according to an X-ray diffraction pattern exhibited by silica, for example, by silica **gel**, one of the conventional porous supports, no distinct diffraction peak can be observed. The X-ray diffraction peaks imply that there is a cyclic structure which exhibits the d-spacing corresponding to the peak angle, in a substance. Therefore, silica **gel** does not have a cyclic structure at least in the range of $d=0.15-12$ nm which is equivalent to 0.7 (deg.) $<2\theta<60$ (deg.). This fact indicates that silica **gel** is amorphous material. Contrary to silica **gel**, the porous substance employed by the present adsorption pump exhibits an X-ray diffraction pattern in which one or more peaks. . . .

DETD Thus, the conventional silica **gel** has such an irregular structure, and accordingly the pores involved in the irregular structure exhibits a heterogeneous pore diameter distribution.. . . .

DETD A process for synthesizing a **meso-porous** substance out of a layer silicate will be hereinafter described. For example, the layer silicate can preferably be kanemite ($\text{NaHSi}.\text{sub.}2$). . . .

DETD . . . it may not be stirred. In addition, it is not necessary to control the pH of the dispersion. However, a **meso-porous** substance of high crystallinity and heat-resistance is preferably prepared in the following manner: namely; first, the pH of the dispersion. . . . further heated. After the heating operation of the dispersion is completed, the resultant solid product is collected by filtering. A **meso-porous** substance of higher heat-resistance is prepared by washing the solid product with water repeatedly. After the washed solid product is. . . . a hydrochloric acid-ethanol mixture solution. This removes the surfactant from the crystals of the solid product to obtain a pure **meso-porous** substance. Note that the calcining operation is preferably carried out by heating the solid product in air, oxygen atmosphere, or. . . .

DETD As having been described so far, the present adsorption heat pump employs a novel adsorbent (e.g., a novel **meso-porous** substance) whose pores have a pore diameter falling in a predetermined range and exhibit a prescribed pore diameter distribution. The novel **meso-porous** substance shows such a large pumping heat quantity (e.g., one of the performance indexes as an adsorbent for adsorption heat pumps) that an adsorption heat pump can exhibit a large pumping temperature difference. Moreover, the novel **meso-porous** substance is also good in terms of the temperature

difference required for regeneration. Therefore, the novel **meso-porous** substance enables a heat source of a low temperature and a small temperature difference to operate adsorption heat pumps. Thus, from this viewpoint as well, the novel **meso-porous** substance is a useful adsorbent for adsorption heat pumps.

DETD **Meso-Porous** Substance Preparation No. 1

DETD . . . 450.degree. C. for 3 hours, and was thereafter calcined in air at 550.degree. C. for 6 hours to prepare a **meso-porous** substance "FSM/16".

DETD Moreover, 4 **meso-porous** substances "FSM/8", "FSM/10", "FSM/12", and "FSM/14" were prepared in the same manner. In the preparation of these extra 4 **meso-porous** substances, instead of the hexadecyltrimethylammonium chloride, the following alkyltrimethylammonium (C._{sub.n} H._{sub.2n+1} N(CH._{sub.3})._{sub.3}) chloride or bromide was used: namely; alkyltrimethylammonium (C._{sub.n} . . atoms was 8, 10, and 12 (i.e., n=8, 10, and 12) in the alkyl group, was used to prepare a **meso-porous** substance "FSM/8", "FSM/10", and "FSM/12", respectively, and alkyltrimethylammonium (C._{sub.n} H._{sub.2n+1} N(CH._{sub.3})._{sub.3}) chloride, in which the number (n) of carbon atoms was 14 (i.e., n=14) in the alkyl group, was used to prepare a **meso-porous** substance "FSM/14". Thus, the resultant 5 **meso-porous** substances were identified with the number (n) of carbon atoms in the alkyl group of the employed surfactant (i.e., alkyltrimethylammonium).

DETD **Meso-Porous** Substance Preparation No. 2

DETD **Meso-porous** substances "FSM/M05", "FSM/M10", and "FSM/M20" were prepared under the similar conditions to those of **Meso-Porous** Substance Preparation No. 1 except for the addition of mesitylene. The mesitylene was added in the hexadecyltrimethylammonium chloride aqueous solution, . . . and mesitylene. An amount of mesitylene added was 0.05M, 0.1M, and 0.2M per 0.1M hexadecyltrimethyl ammonium chloride to prepare the **meso-porous** substances "FSM/M05", "RFSM/M10", and "FSM/M20", respectively. Thus, the resulting 3 **meso-porous** substances were identified with the concentration of the employed mesitylene aqueous solutions.

DETD (Structural Analysis on **Meso-Porous** Substances)

DETD The thus synthesized powder of the **meso-porous** substances was subjected to an X-ray diffraction analysis, and to a transmission-electron-microscope analysis, and the resultant X-ray diffraction patterns and. . .

DETD As can be seen from the X-ray diffraction patterns illustrated in FIG. 7, the **meso-porous** substances "FSM/12", "FSM/14", "FSM/16", and "FSM/M05" were observed to exhibit three to four diffraction peaks in a diffraction-angle range of 10 deg. or less. The peaks could be indexed to a hexagonal lattice. Whilst, the **meso-porous** substances "FSM/8", "FSM/10", and "FSM/M10" were observed to exhibit one to two diffraction peaks in a diffraction-angle range of 10 deg. or less. Moreover, the **meso-porous** substance "FSM/M20" was not observed to exhibit any appreciable peaks in a diffraction-angle range of 1 deg. or more. According to these results of the analysis on these X-ray diffraction patterns, these **meso-porous** substances were found to have a regular structure.

DETD FIG. 8 is a photograph of the **meso-porous** substance "FSM/16" taken by the transmission electron microscope, and shows the pore diameter distribution in the **meso-porous** substance "FSM/16". It is evident from the photograph that pores having a pore diameter of 2.8 nm were arranged regularly. . .

DETD (Determination of Pore Diameter Distribution Curve of **Meso-Porous** Substances)

DETD The pore diameter distribution curves exhibited by the aforementioned

meso-porous substances were determined in accordance with a nitrogen adsorption isotherm. The nitrogen adsorption isotherm was measured in the following manner. . . . be connected with the vacuum line via one of the control valves. In the calibration, a sample of the aforementioned **meso-porous** substances was weighed out by about 40 mg, and was put in a sample tube made of glass. The sample. . . . the adsorption amounts derived from the pressure variation were plotted to prepare a nitrogen adsorption isotherm for each of the **meso-porous** substances. FIG. 9

illustrates the results of the nitrogen-adsorption-isotherm measurement.

DETD Based on the nitrogen adsorption isotherms shown in FIG. 9, pore diameter distribution curves of the aforementioned 8 **meso-porous** substances were determined by the Cranston-Inklay method. FIG. 10 illustrates the results of the determination. The following physical properties were. . . . distribution curves (hereinafter referred to as a "central pore diameter"), a total volume of all the pores involved in the **meso-porous** substances, and a proportion of a summed volume of pores, having a pore diameter falling in a range of .+-40%. . . .

DETD		. . .	0.85	80
	FSM/16	2.8	1.04	64
No. 2	FSM/M05	3.2	1.03	68
	FSM/M10	3.6	1.20	63
	FSM/M20	4.7	1.22	60
Comp.	Silica Gel			
		2.3	0.46	35
Ex.	Activated			
		2.3	0.63	56
	Carbon			
	Zeolite	0.5	0.16	>90

DETD It is apparent from Table 1 that all of the **meso-porous** substances prepared by Preparation Nos. 1 and 2 had a central pore diameter which fell in a range of from. . . .

DETD Moreover, as comparative examples, a nitrogen adsorption isotherm was calibrated for a commercially available type "A" silica **gel**, an activated carbon "Kurare D7", and a zeolite "ZSM-5" in the aforementioned manner. FIG. 11 illustrates the resultant nitrogen adsorption. . . .

DETD It is understood from Table 1 that the silica **gel** and activated carbon had a central pore diameter which fell in the range specified by the present invention, but that. . . .

DETD (Densifying **Meso-Porous** Substances)

DETD packing density of porous substances will be hereinafter described. For instance, in this Second Preferred Embodiment, a powder of a **meso-porous**-substance powder was pressurized by the cold isostatic press (CIP).

DETD As test specimens, the same **meso-porous** substances "FSM/10" and "FSM/16" as prepared in Preparation No. 1 of the First Preferred Embodiment were also employed. Note that, however, the **meso-porous** substances "FSM/10" and "FSM/16" of the Second Preferred Embodiment were those prior to the final stage of Preparation No. 1 of the First Preferred Embodiment. Specifically, the **meso-porous** substances "FSM/10" and "FSM/16" of the Second Preferred Embodiment were not subjected to the final heating and calcination processes which. . . . in air, respectively. Thus, the organic surfactants (i.e., octadecyltrimethylammonium bromide, and hexadecyltrimethylammonium chloride) were resided in the pores of the **meso-porous** substances "FSM/10", and "FSM/16", respectively.

DETD density thereof was calculated. Table 2 below sets forth the packing densities of the test specimens made by pressurizing the **meso-porous** substance "FSM/10" at various pressures.

Whilst, Table 3 below sets forth the packing densities of the test specimens made by pressurizing the **meso-porous** substance "FSM/16" at various pressures.

DETD It is appreciated from Table 2 that the test specimen made by pressurizing the **meso-porous** substance "FSM/10" at 5,000 kgf/m.² exhibited a packing density of 0.78 g/ml, which was more than twice that of the . . . to the pressing operation. FIG. 13 illustrates a pore diameter distribution curve of the test specimen made by pressurizing the **meso-porous** substance "FSM/10" at 5,000 kgf/m.², and a pore diameter distribution curve of the test specimen made by pressurizing the **meso-porous** substance "FSM/16" at 6,000 kgf/m.². Thus, the test specimens were verified to exhibit a sharp pore diameter distribution curve, respectively.

DETD In addition, the test specimen, made by pressurizing the **meso-porous** substance "FSM/10" at 5,000 kgf/m.², was found to exhibit a central pore diameter of 1.9 nm, and a .+- .40% pore proportion of 71%. The test specimen, made by pressurizing the **meso-porous** substance "FSM/16" at 6,000 kgf/m.², was found to exhibit a central pore diameter of 2.8 nm, and a .+- .40% pore. . . .

DETD . . . water-vapor adsorbing apparatus, "BELSORP 18", made by NIHON BELL Co., Ltd. In this determination of the water-vapor adsorption isotherms, the **meso-porous** substance "FSM/10" was pressed at 5,000 kg m.², and was screened to have a particle diameter of from 0.25 to 0.5 mm. FIG. 14 illustrates the water-vapor adsorption isotherms exhibited by the **meso-porous** substance "FSM/10". The **meso-porous** substance "FSM/16" was pressed at 6,000 kgf/m.², and was screened to have a particle diameter of from 0.25 to 0.5 mm. FIG. 15 illustrates the water-vapor adsorption isotherms exhibited by the **meso-porous** substance "FSM/16". For comparison, a commercially available type "A" silica **gel** having a particle diameter of from 0.1 to 0.15 mm was similarly examined for the water-vapor adsorption isotherms. FIG. 16 illustrates the water-vapor adsorption isotherms exhibited by the type "A" silica **gel**.

DETD As can be seen from FIGS. 14 and 15, the test specimens, made from the **meso-porous** substances "FSM/10" and "FSM/16", showed a large hysteresis in their adsorption-desorption isotherms during the first cycle of the water-vapor-adsorption-isotherm determination. . . . This phenomenon is believed to have resulted from the hydration occurred on the surface of the test specimens (i.e., the **meso-porous** substances "FSM/10" and "FSM/16") during the adsorption in the first cycle. After the second cycle, they exhibited a substantially identical. . . .

DETD On the other hand, as illustrated in FIG. 16, a comparative test specimen, the type "A" silica **gel**, exhibited a substantially identical adsorption-desorption isotherm during the first, second and third cycles of the water-vapor-adsorption-isotherm determination. . . .

DETD . . . a type "C" structural activated carbon, a type "D" structural activated carbon, a molecular-sieve carbon, and a type "B" silica **gel**. Accordingly, the same data, such as the maximum variations of adsorption, etc., were also calculated from the disclosed water-vapor adsorption. . . .

DETD . . . (g/ml)

2nd Pref.

FSM/10 0.78
0.2-0.4 0.25 0.20

Embodiment

FSM/16 0.67
0.4-0.6 0.44 0.29

Comp. Ex.

Type "A"

0.70			
0.4-0.6	0.14	0.10	
Silica Gel			
Type "A"			
0.263			
0.3-0.5	0.12	0.032	
S.A.C.*			
Type "B"			
0.241			
0.3-0.5	0.16	0.039	
S.A.C.*			
Type "C"			
0.121			
0.2-0.4	0.21	0.025	
"D"			
0.255			
0.5-0.7	0.58	0.15	
S.A.C.*			
Molecular-			
0.348			
0.4-0.6	0.14	0.049	
Sieve Carbon			
Type "B"			
--	0.0-0.2	0.15	--
Silica Gel			

Note (1): The data on the comparative examples marked with * are excerpted from the aforementioned literature.

Note (2): The. . .

DETD It is evident from Table 4 that the **meso-porous** substances "FSM/10" and "FSM/16", the novel adsorbents employed by the present adsorption heat pump, exhibited a superb maximum variation of. . . could be considered a good adsorbent for adsorption heat pumps. Whilst, the actual comparative example (e.g., the type "A" silica **gel**), and the comparative examples excerpted from the literature (e.g., the type "A" structural activated carbon, and so on) exhibited an.

DETD . . . with the calculation method disclosed in the aforementioned literature, a pumping heat quantity and a pumping temperature difference of the **meso-porous** substances "FSM/10" and "FSM/16" employed by the present adsorption heat pump were determined. The results of this determination are summarized. . .

DETD . . . Pref.

FSM/16	0.40		
	0.60		
	0.18		
	0.44		
		286	8.6 11.5

Emodi-
ment
Comp. Ex.

Type "A"			
0.10			
0.70			
0.18			
0.34			
	238	6.1	7.9

Silica Gel			
Type "A"			
0.22			
0.58			
0.020			
0.153			

40 9.2 11.5

S.A.C.*

Type "B"

0.22

0.57

0.045

0.199

48 10.8

12.6

S.A.C.*

Molecular-

0.39

0.62

0.002

0.191

66 9.5 11.9

Sieve

Carbon

Type "B"

0.21

0.85

0.155

0.148

-- 4.0 4.2

Silica Gel

Note (1): The data on the comparative examples marked with * are excerpted from the aforementioned literature.

Note (2): The. . .

DETD It is apparent from Table 4 that the **meso-porous** substances "FSM/10" and "FSM/16", the novel adsorbents employed by the present adsorption heat pump, exhibited a large pumping heat quantity. . .

DETD For example, the pumping temperature differences exhibited by the **meso-porous** substance "FSM/10" of the Second Preferred Embodiment is compared with those exhibited by the type "A" silica **gel** and the type "D" structural activated carbon of the comparative examples. The **meso-porous** substance "FSM/10" exhibited a pumping temperature difference $\Delta T_{sub.c} = 15.9$ K, and a pumping temperature difference $\Delta T_{sub.h} = 22.5$ K. On the other hand, the type "A" silica **gel** exhibited a pumping temperature difference $\Delta T_{sub.c} = 6.1$ K, and a pumping temperature difference $\Delta T_{sub.h} = 7.9$ K. The type "D" structural. . . difference $\Delta T_{sub.c} = 6.8$ K, and a pumping temperature difference $\Delta T_{sub.h} = 8.4$ K. Thus, the pumping temperature differences exhibited by the **meso-porous** substance "FSM/10" were twice as much as, or more of those exhibited by the type "A" silica **gel** and the type "D" structural activated carbon. Thus, the **meso-porous** substance "FSM/10" employed by the present adsorption heat pump exhibited a large pumping heat quantity, and a large pumping temperature difference, both of which were well balanced with each other. Therefore, the **meso-porous** substance "FSM/10" apparently made a good adsorbent for adsorption heat pumps.

CLM What is claimed is:

5. The adsorption heat pump according to claim 4, wherein said silicon oxide is a **meso-porous** substance.

6. The adsorption heat pump according to claim 5, wherein said **meso-porous** substance is a **meso-porous** molecular sieve.

7. The adsorption heat pump according to claim 5, wherein said

meso-porous substance is produced from a dispersion which includes a layer silicate and a surfactant.

11. The adsorption heat pump according to claim 7, wherein said **meso-porous** substance produced from the dispersion is further calcined in air, oxygen atmosphere, or nitrogen atmosphere at a temperature of 50.degree.. . .

L8 ANSWER 11 OF 12 USPATFULL
AN 97:84013 USPATFULL
TI Process for preparing silicon carbide foam
IN Whinnery, LeRoy Louis, Livermore, CA, United States
Nichols, Monte Carl, Livermore, CA, United States
Wheeler, David Roger, Albuquerque, NM, United States
Loy, Douglas Anson, Albuquerque, NM, United States
PA Sandia Corporation, Albuquerque, NM, United States (U.S. corporation)
PI US 5668188 19970916
AI US 1996-586453 19960116 (8)
DT Utility
FS Granted
LN.CNT 542
INCL INCLM: 423/345.000
INCLS: 264/044.000; 264/051.000; 264/029.100; 264/029.700; 423/345.000;
423/445.000; 423/449.000; 501/088.000; 521/918.000; 521/064.000;
502/101.000; 502/180.000; 502/416.000; 502/418.000
NCL NCLM: 423/345.000
NCLS: 264/029.100; 264/029.700; 264/044.000; 264/051.000; 501/088.000;
502/101.000; 502/180.000; 502/416.000; 502/418.000; 521/064.000;
521/918.000
IC [6]
ICM: C08J009-28
EXF 521/64; 521/918; 423/345; 423/445; 264/44; 264/29.7; 264/51; 264/29.1;
501/88
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB A method of preparing near net shape, monolithic, porous SiC foams is disclosed. Organosilicon precursors are used to produce polymeric gels by thermally induced phase separation, wherein, a sufficiently concentrated solution of an organosilicon polymer is cooled below its solidification temperature to form a **gel**. Following solvent removal from the **gel**, the polymer foam is pretreated in an oxygen plasma in order to raise its glass transition temperature. The pretreated foam is then pyrolyzed in an inert atmosphere to form a SiC foam.
AB . . . phase separation, wherein, a sufficiently concentrated solution of an organosilicon polymer is cooled below its solidification temperature to form a **gel**. Following solvent removal from the **gel**, the polymer foam is pretreated in an oxygen plasma in order to raise its glass transition temperature. The pretreated foam. . .
SUMM . . . ability to tailor the internal structure (porosity) of the SiC foam and, in particular, they cannot be used to produce **meso-porous** SiC foams which for the purposes of the instant invention is defined as having pores which are about 10-100 .mu.m. . .
SUMM . . . phase separations (TIPS) wherein a sufficiently concentrated solution of a polymer is cooled below its transition temperature to form a **gel**.
DRWD . . . A polysilane polymer is first dissolved in an appropriate solvent, preferable cyclohexane. The solution is next cooled unidirectionally to promote **gel** formation as described in U.S. Pat. No. 4,673,695, incorporated herein by reference. The solvent can then be removed from the **gel** either by a freeze drying process (sublimation under vacuum) or by supercritical CO₂ extraction leaving behind a polysilane foam. However,. . .
DRWD . . . a plasma treatment step, preferably the use of an oxygen plasma

DRWD treatment step, to be useful to stabilize polysilane polymer **gel** foams. The polysilane foams are placed in a radio frequency generator for a predetermined length of time depending generally upon. . . . rapid one-dimensional cooling to temperatures in the range of -10 to +50 C to induce phase separation, thereby forming a **gel**. Solvent is removed from the **gel** to form a polysilane foam. The step of solvent removal can be by freeze drying or by supercritical CO₂ extraction.. . .

DRWD . . . the polymer phase being retained by cooling the system at a rate sufficient to freeze the polymer, thereby forming a **gel**, before a change in structure can occur. To produce anisotropic foams, the solution of polymer and solvent are selected such. . .

DRWD . . . another extraction methods known to those skilled in the art, for example, supercritical CO₂ extraction. In either instance, a polymer **gel** having a continuous, monolithic foam structure remains. The foam that remains after the solvent has been removed from the **gel** will retain the shape of the container or mold into which the solution had been poured. By allowing for shrinkage of the **gel** foam during subsequent processing steps and sizing the mold accordingly, it is possible to use the instant invention to produce. . .

DETD . . . In order to eliminate bubble formation in the final product it was necessary to remove any residual gasses from the **gel**. A top, fitted with a septum, was used to evacuate the sample. To prevent headspace vapor cloud condensation and subsequent. . .

CLM What is claimed is:

. . . dissolving an organosilicon polymer or copolymer in a solvent to form a solution; b) cooling the solution to form a **gel**; c) removing the solvent from the **gel** to form a polymer foam; d) stabilizing the polymer foam by subjecting the foam to a plasma; and e) pyrolyzing. . .

L8 ANSWER 12 OF 12 USPATFULL
AN 89:9295 USPATFULL
TI Heterogeneous catalyst containing silicon dioxide
IN Aulich, Hubert, Munich, Germany, Federal Republic of
Urbach, Hans-Peter, Munich, Germany, Federal Republic of
Eisenreith, Karl-Heinz, Schliersee, Germany, Federal Republic of
PA Siemens Aktiengesellschaft, Berlin and Munich, Germany, Federal Republic
of (non-U.S. corporation)
PI US 4803188 19890207
AI US 1988-142203 19880111 (7)
PRAI DE 1987-3702363 19870127
DT Utility
FS Granted
LN.CNT 234
INCL INCLM: 502/232.000
INCLS: 502/408.000; 502/439.000
NCL NCLM: 502/232.000
NCLS: 502/408.000; 502/439.000
IC [4]
ICM: B01J037-00
ICS: B01J021-06
EXF 502/3; 502/408; 502/439; 502/232
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The present invention provides a heterogeneous catalyst based on silicon dioxide, as well as, a method for manufacturing same. The catalyst of the present invention is constructed from a porous material produced from large-surface glass bodies containing catalytically active additives by leaching with mineral acid and the additives are incorporated into the network of the glass body in the form of metal oxides insoluble in hot mineral acid. The catalyst exhibits a high active surface and is universally employable because it contains only a

SUMM slight amount of undesired impurities, is highly reactive, and has a high structural stability up to about 1000.degree. C.

SUMM Naturally occurring, porous materials, such as, for example, diatomaceous earth, bentonites, silica gel, pumice, aluminum silicates, zeolites, or aluminum oxide, have been used as catalysts for some fields of application. These materials are. . .

SUMM . . . a low content of impurities, specifically an alkali content below 0.1 weight ppm. Furthermore, the resultant catalyst is a stable **meso-porous** structure that is largely preserved up to 1000.degree. C.

CLM What is claimed is:

1. A heterogeneous catalyst based on silicon dioxide wherein the catalyst is a **meso-porous** material produced from large-surface glass bodies containing catalytically active additive by leaching with mineral acid, the catalytically active additives including. . .
- . . . glass melt; (d) subjecting the glass bodies to a leaching process in hot mineral acid; and (e) washing the resultant **meso-porous** material.

L4 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2001 ACS
AN 1999:93076 CAPLUS
TI Matrix effects on electrochemical reactions in **sol-gel** materials
AU Cox, James A.
CS Department of Chemistry and Biochemistry, Miami University, Oxford, OH,
45056, USA
SO Book of Abstracts, 217th ACS National Meeting, Anaheim, Calif., March
21-25 (1999), PHYS-073 Publisher: American Chemical Society, Washington,
D. C.
CODEN: 67GHA6
DT Conference; Meeting Abstract
LA English

=> d L4 1 kwic

L4 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2001 ACS
TI Matrix effects on electrochemical reactions in **sol-gel** materials
AB **Sol-gel** materials are being evaluated as electrolytes for
electrochem. in the absence of a contacting liq. phase. The voltammetry
of hosted. . . Deff varies over the range 10-8 to 10-6 cm²s⁻¹ as the
silica is templated to change from a microporous to **mesoporous**
material. With polymeric dopants such as Prussian Blue, the
values remain in the range 10-8 cm²s⁻¹. These data reflect the relative.
. . pathways in silica will be shown by measurement of the rate of
disproportionation of UO₂⁺. How these results allow optimizing
doped gels as platforms for sensors and as electrocatalysts for
redox of gaseous species will be discussed.

L5 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2001 ACS
 AN 1993:83956 CAPLUS
 DN 118:83956
 TI Stabilization of synthetic mesoporous crystalline material, and the material obtained
 IN McCullen, Sharon B.; Vartuli, James C.
 PA Mobil Oil Corp., USA
 SO U.S., 21 pp. Cont.-in-part of U.S. Ser. No. 625,245.
 CODEN: USXXAM
 DT Patent
 LA English
 IC ICM C01B033-34
 NCL 423718000
 FAN.CNT 13

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 5156829	A	19921020	US 1991-718505	19910620
	US 5102643	A	19920407	US 1990-470008	19900125
	US 5098684	A	19920324	US 1990-625245	19901210
PRAI	US 1990-470008	A2	19900125		
	US 1990-625245	A2	19901210		
OS	MARPAT 118:83956				
AB	. . . with a compn. comprising $M'X_2Y_n$ (M' is .gtoreq.1 of Ti, B, Al, and Si; X is .gtoreq.1 of C1-6-halides and -alkoxides ; Y is .gtoreq.1 of X and C1-12-alkyls; n = 1-2) under conditions to yield a stabilized cryst. material. The cryst. . . .				
IT	Silica gel , reactions				
	RL: RCT (Reactant) (reactions of, in cryst. mesoporous material manuf., HiSil)				
IT	505-86-2, Cetyltrimethylammonium hydroxide				
	RL: PROC (Process) (reactions in presence of, in cryst. mesoporous material manuf.)				
IT	681-84-5, Tetramethyl orthosilicate 2269-22-9, Aluminum tri-sec-butoxide 3087-36-3, Titanium tetraethoxide 3453-79-0, Aluminum tri-isobutoxide				
	RL: RCT (Reactant) (reactions of, hydrothermal, in cryst. mesoporous material manuf.)				
IT	53116-81-7, Tetramethylammonium silicate				
	RL: RCT (Reactant) (reactions of, in cryst. mesoporous material manuf.)				
IT	24623-77-6P, Alumina monohydrate				
	RL: PREP (Preparation) (.alpha.-, reactions of, in cryst. mesoporous material manuf., Catapal)				
AB	The process comprises contacting the material having x-ray diffraction pattern having .gtoreq.1 peaks at d-spacing .gtorsim.18 .ANG. and C6H6 adsorption capacity >15 g/100 g at 50 torr and 25.degree., with a compn. comprising $M'X_2Y_n$ (M' is .gtoreq.1 of Ti, B, Al, and Si; X is .gtoreq.1 of C1-6-halides and -alkoxides ; Y is .gtoreq.1 of X and C1-12-alkyls; n = 1-2) under conditions to yield a stabilized cryst. material. The cryst. material has compn. (anhyd. basis) $rRMn/q(WaXbYcZdOh)$ [R = total org. material not included in M; r = no. of moles or mole fraction of R; M is .gtoreq.1 ions; n = charge of the compn. excluding M expressed as oxides: q = weighted mol. av. valence of M; n/q no. of moles or mole fraction of M; W is .gtoreq.1 divalent elements; X is .gtoreq.1 trivalent elements; Y is .gtoreq.1 tetravalent elements; Z is .gtoreq.1 pentavalent elements; a, b, c, and d = mole fraction of W, X, Y, and Z, resp., h = 1-2.5; (a + b + c + d) = 1]. A mixt. of cetyltrimethylammonium hydroxide 40, Catapal (.alpha.-Al2O3 monohydrate)				

0.8, tetramethylammonium silicate 20, HiSil (pptd. SiO₂ contg. 6 wt.% free water and 4.5 wt.% bonded water; particle size 1 to eq. 0.02 μ m) 5, and cetyl alc. was crystd. under agitation at 105.degree. for 4 h, calcined at 538.degree. in N for 2 h and in air for 2 h, mixed with 1 g (EtO)₄Si, 5 g deionized water, allowed to react for 1 h, and the solid product was heated at 538.degree. in N for 2 h and in air for 2 h to give a cryst. product having C₆H₆ adsorption 400 mg/g at 30.degree..